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YAG:Er,Yb	YAG:Ho	

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report describes the single crystal growth, laser rod fabrication and experiments on about 25 rare earth doped YAG systems. The objective was to examine specified compositions for possible lasing in the 1.5-4.0μm range. Since a mature technology base is already available for Nd:YAG, any of the the newly developed materials can be put in rapid production if necessary. The particular crystals were prepared with Er, Dy, Ho, Tm, Tm-Cr,

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Pr-Cr, Yb, Eu, and U dopants. Various concentrations of dopants were utilized. Large crystals were grown to extract up to 5 x 50mm laser rods. Different fabricated shapes were provided for spectroscopic studies or pumping by other lasers. Special coatings for new wavelengths were applied to permit optimum laser results. The final active testing was performed on each item at the Naval Research Laboratory. Test results are not incorporated in this report.

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FOREWORD

This Final Report describes the single crystal growth of rare earth doped YAG laser crystals. The major items are directly concerned with specific rare earth doped Er, Tm, Ho, Yb, Eu which may prove to be suitable for possible new lasing schemes in the 2-5μm range. Another portion of the effort was directed towards the growth and laser fabrication of special YAG crystals. The report summarizes all efforts under Contract No. N00173-79-C-0134 for the period May 1, 1979 to October 1, 1981. The contract work was under the coordination of Dr. Van O. Nicolai of the Office of Naval Research.

All compositions, preparations, single crystal growth, and laser rod fabrication and coating were performed in the laboratories of Airtron Division of Litton Systems, Inc., 200 East Hanover Avenue, Morris Plains, New Jersey 07950. Dr. Roger F. Belt was the technical director of the project and Dr. Larry Drafall was the project engineer. Karl Jensen was the senior growth technician. Steven Turner supervised laser material fabrication and provided the coated optics. Active testing of these laser samples was conducted at the Naval Research Laboratory by Dr. Leon Esterowitz. The report was prepared by Dr. Roger F. Belt and released for publication in December 1981.

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1.0 Introduction

At the present time the Nd:YAG laser is still the most widely used and satisfactory from the viewpoint of physics and materials. It would be of great interest to extend the emitting wavelengths beyond the 1.06μm of Nd to 1.5-4.0μm and utilize the YAG host simultaneously. A cursory look at energy levels of the individual rare earth ions in YAG shows that this is possible only with certain elements, viz. Dy, Er, Tm and Ho. Another approach which has not been attempted in YAG but has been in fluorite hosts, viz. U^{3+} in CaF_2 may be worthwhile to pursue. A good review of all the early solid state laser crystal development may be found in the extensive book by Kaminskii.(1) Laser action is indeed possible up to about 3.91μm when Ho^{3+} in $YLiF_4$ is used. Most of these systems must be operated at low temperatures but there are several lasers capable of working at 300K. Good examples are Er in YAG and Ho in $YLiF_4$. The former has been developed more in Russia by various laser groups(2,3) while the latter by various workers in the United States.(4)

One of the necessary conditions for extending laser investigations is good cooperation between crystal growers and laser physicists. High quality materials must be made available in order to examine physically possible energy level transitions for laser action. The present small program

was initiated by workers at the Naval Research Laboratory to examine and prepare a list of carefully selected materials based on the YAG host. Some of these may have been prepared before but all of our crystals were grown for the purpose of yielding laser rods equivalent to production Nd:YAG. In general the dopants were chosen mainly from those ions capable of laser action in the 1.5-4.0μm range. Several consultations were held to choose the final dopant level, proposed valence state, or other problems encountered in the growth process. The laser rod configuration was chosen carefully along with the end face coatings necessary for the sought laser action.

The doping of our crystals did not bring any special problems because most of the intended ions had a size very close to that of yttrium.[5] Thus their distribution coefficients were very close to unity. In fact we grew boules of YAG with up to 5 or 10% substitution with no difficulty or impairment of quality. In the case of Er the completely substituted garnet $Er_3Al_5O_{12}$ was grown. One totally new system was examined which presented certain problems in control of valence state. This was the attempt to grow U^{3+} in YAG.

2.0 Experimental

The growth of large single crystals of doped $Y_3Al_5O_{12}$ (YAG) was performed on one of Airtron's production type Czochralski growth stations. A station is illustrated in

Figure 1. Iridium crucibles of 2 or 2.5 inch diameter were used to contain the melt and to serve as a susceptor for the 450 KHz RF heating. The crucibles were contained within a glass bell jar in order to maintain a controlled atmosphere. A mixture of N_2-O_2 was used for oxidizing conditions and one of N_2-H_2 for reducing conditions. The latter atmosphere was mainly for valence control in YAG:U^{3+}. All crystals were doped with various rare earths in a range of 0.05 to 10%. Appropriate segregation coefficients were applied for each doping element.[5] Total crystal lengths of 3-4 inches and diameters of 0.6-0.8 inch were grown. These boules were sufficient to provide a (6 x 60)mm laser rod plus additional pieces for sizeable polished rectangular blocks, cubes, or discs. The latter were used for many experiments in spectroscopy or as direct samples pumped by other lasers.

All crystals were grown along [111]. Seeds were composed of undoped YAG to prevent contamination of the melt with traces of other elements such as Nd. It was also necessary to clean the crucibles thoroughly between runs to avoid trace contamination from the various dopants. Cleaning was performed by core drilling and removing most of the residual melt, flux treatment to dissolve material, and finally a regular acid cleaning. Crucibles are then preheated, checked for leaks, and repaired if necessary before the next crystal growth run.

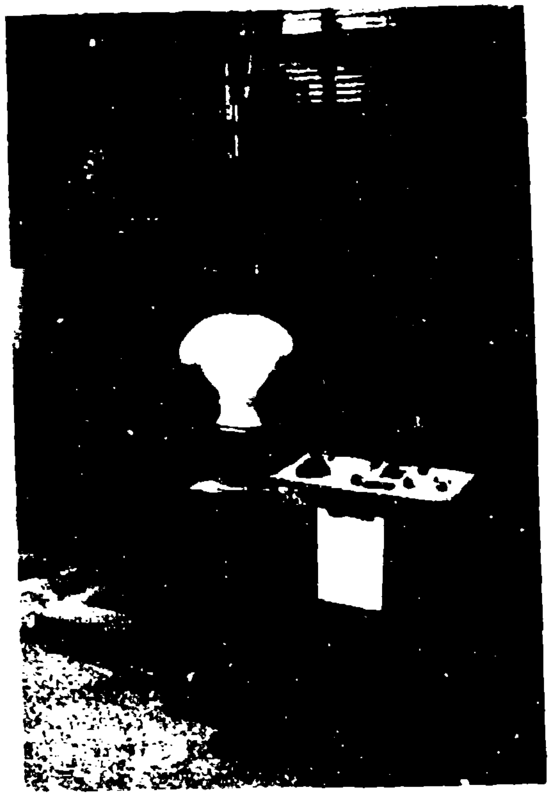

Figure 1   Czochralski Growth Station

Laser rods were fabricated in Airtron's production facility and presented no special problems. Several special end configurations such as brewster angle, off orientation, and convex curvature were prepared in addition to the flat-flat type. Rectangular parallelepipeds also were fabricated where pumping was performed with another laser instead of a flashlamp. Most of the finished rods were prepared with single layer AR coatings for the chosen wavelength of testing. However a number of rods required special multilayer coatings for dual wavelengths in the 1.5-4μm range. These rods were not coated at Airtron but were done at Optical Filter Corp. of Natick, Mass.

3.0 Results

At the beginning of this program a list of the desirable laser rods was formulated and agreed upon. Some of these compositions were grown previously and others were not. Therefore from a crystal growth viewpoint it was decided that the best procedure would be to progess from the easiest to the more difficult. Usually this meant that the YAG crystals with the lowest doping level were tried; then we proceeded to high dopants. Finally those crystals with special problems of doping, valence control, or poor quality were grown when more time could be spent on runs. Otherwise the runs and their numbers described in the following paragraphs and tables have no significance.

3.1 Crystals with Low Dopant Levels

The first crystal attempted was a 2% Er doped YAG. The low dopant level and size of the Er suggested a station arrangement similar to that of pure YAG. A good crystal was grown at a pull rate of about 2mm/hr and a rotation rate of 15 rpm. During the cooling program and after 15cm of crystal was grown, a crack initiated from the side. When the boule was removed and examined, the crystal quality was good except for some slight scattering near the last 2cm of the boule. A picture of the crystal from run NC-51 is given in Figure 2. Sufficient material of hig quality enabled us to get laser rods.

The next crystal attempted was a 2% dy in YAG. The crystal was grown at a pull rate of 1.2mm/hr and a rotation rate of 30 rpm. Two trials were made on the composition. In the first a small malfunction occurred in the RF generator and a diameter excursion arose which gave a cracked crystal at the top. No rods could be extracted from the boule. The second attempt gave a very good crystal which is shown in Figure 3 from run NC-54-2.

Growth run NC-55 was designed as a 2% Ho in YAG. This run was made with a minimum of difficulty and gave a good crystal at a pull rate of 1.2mm/hr and rotation rate of 30 rpm. Rods and cubes were extracted. The boule had a few bubbles and a slight amount of scattering in certain areas.

Figure 2 YAG - 2% Er from NC-51

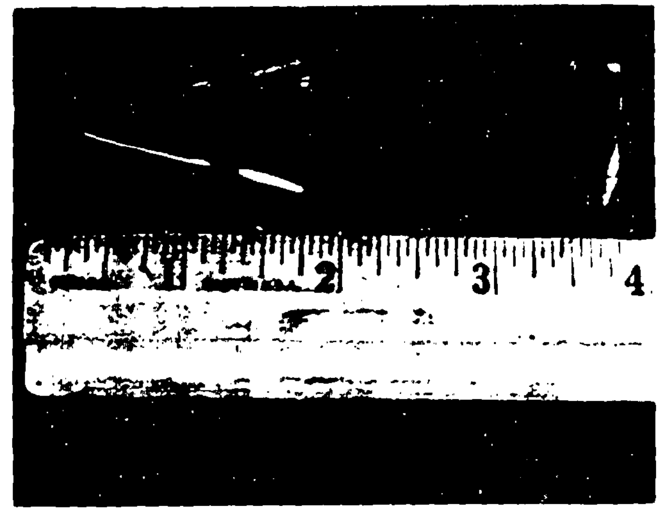

Figure 3 YAG - 2% Dy from NC-54-2

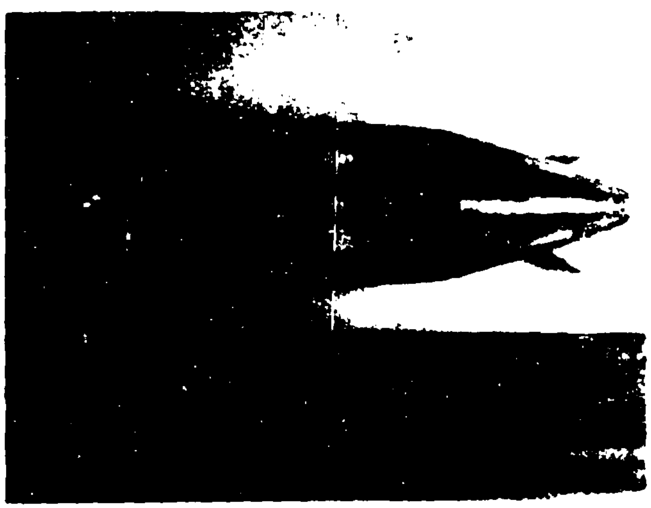

Figure 4 YAG - 2% Ho from NC-55

Figure 5 YAG - 2% Ho from NC-55-2

The crystal is pictured in Figure 4. Through melt replenishment another crystal was grown at the same Ho level but at a higher growth rate of 2.5mm/hr. This crystal from run NC-55-2 gave an excellent boule and is shown in Figure 5.

At this stage in the program the crucible size was switched to 2 inch and a crystal of $Er_3Al_5O_{12}$ was grown. The run NC-56 proceeded with no difficulty and an excellent crystal was grown at 1.2mm/hr. The result is shown in Figure 6.

With a 3 inch crucible a new attempt was made with 1% Ho in YAG. This run gave a highly perfect crystal with superb diameter control. A high yield of rods and other shapes could be obtained. Figure 7 shows the crystal. A pull rate of 1.2mm/hr was satisfactory for crystal NC-58. These experiments completed all of the requirements for materials of low dopant levels.

3.2 High Dopant or Double Doped Systems

The first of these crystal growth runs was NC-61 which was prepared as a 5% Tm in YAG. A 2 inch crucible was used and a pull rate of 1.1mm hour at 30 rpm. About 3-4cm of single crystal was grown and the station was shut down by a power excursion. The boule yielded enough single crystal to prepare the requested size and shape of delivered sample. The same melt was also used for a new crystal which was double doped with 10% Tm and 0.5% Cr. This composition was achieved by further additions to NC-61 since only 3-4% of the melt was

Figure 6 $Er_3Al_5O_{12}$ from NC-56

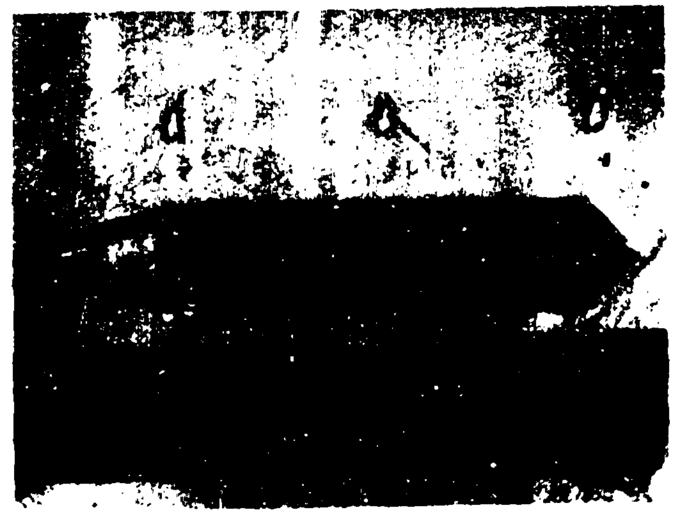

Figure 7 YAG - 1% Ho from NC-58

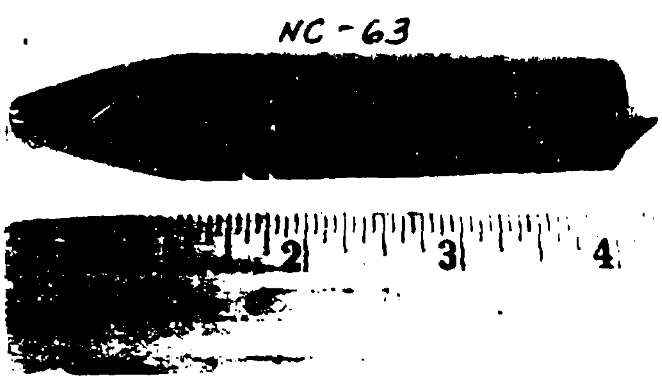

Figure 8 YAG - 10% Tm, 0.5% Cr from NC-63

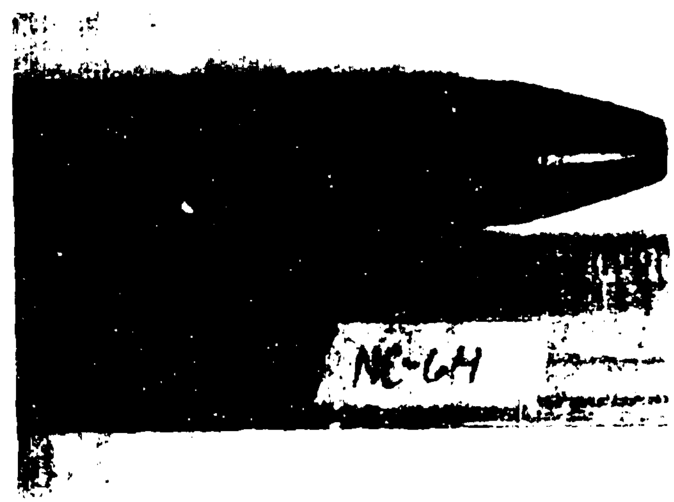

Figure 9 YAG - 1% Cr, 0.5% Pr from NC-64

removed. The double doped run was NC-63 and a crystal was grown at 0.6mm/hr and 30 rpm. This crystal was green in color and is shown in Figure 8. Good diameter control is evident and the internal quality was high. A few inclusions were observed but rods were obtained easily.

The next crystal attempted was a 1.0% Cr and 0.5% Pr doping. The run was NC-64 and made at a growth rate of 0.6mm/hr and 30 rpm. Results are shown in Figure 9. Since both Cr and Pr are green colored ions, the crystal was a fairly dark green. The diameter control, internal quality, and rod yield were all good.

A single run was tried with a 5% doping of Yb. This run from NC-65 gave an excellent crystal and is illustrated in Figure 10. The pull rate and rotation rate were the same as the previous run. Immediately after this experiment a 3% Eu composition was attempted. A good crystal was grown in run NC-66 and is shown in Figure 11. Two difficulties were found with this crystal in spite of the excellent diameter control. Under polarized light a series of bands parallel to (111) planes was found. About midway through the crystal a high area of strain was found along one side of the boule. The bands are thought to be related to a stoichiometry problem involving N_2-O_2 control, a change of valence state of the Eu, or color center (vacancy in the lattice). This boule was heated subsequently in an oxygen atmosphere. The strained

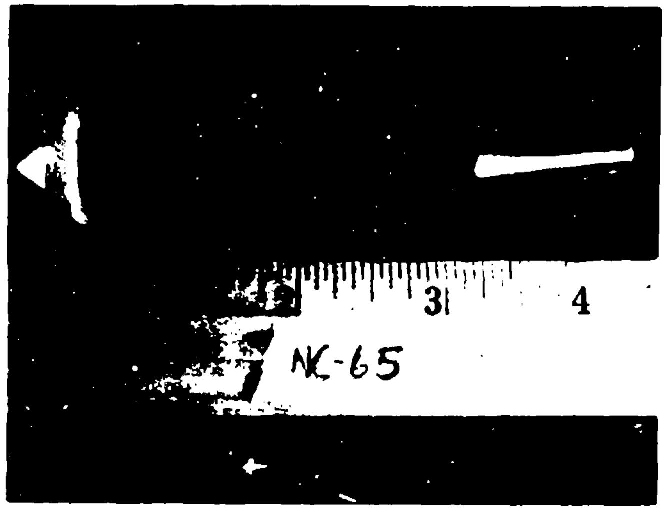

Figure 10 YAG - 5% Yb from NC-65

Figure 11 YAG - 3% Eu from NC-66

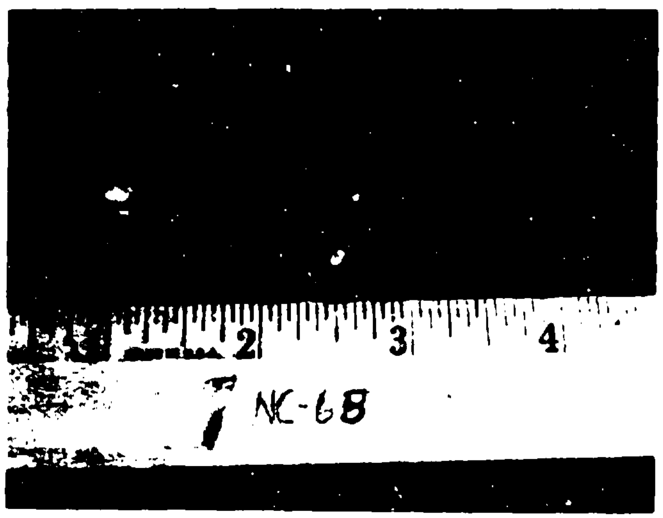

Figure 12 $Er_3Al_5O_{12}$ from NC-68

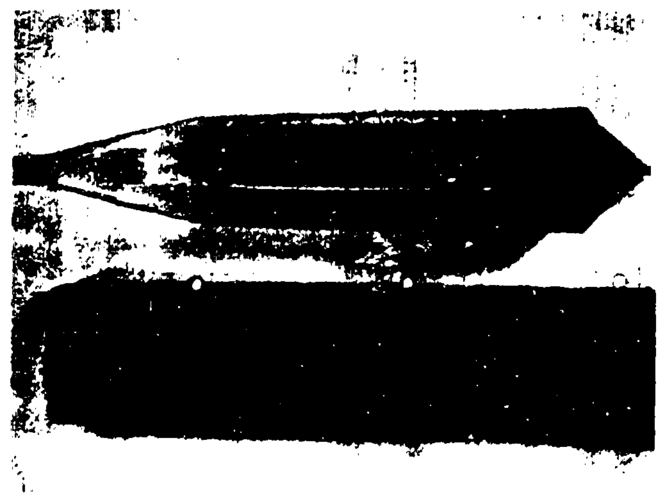

Figure 13 YAG - 0.05% U^{3+} from NC-72

area was not affected but the banding was diminished. For this same melt an adjustment was made in the composition to bring the Eu level to 5%. A new crystal was grown from run NC-67. This boule had excellent diameter control with no strained areas. There was also evidence of light banding as in the previous run. All rods and samples were cut from clear areas.

Since additional material was necessary for $Er_3Al_5O_{12}$ requirements, an additional run was made in a 2 inch iridium crucible. This run was NC-68 and is pictured in Figure 12. The pull rate was 0.6mm/hr and rotation rate was 30 rpm. Diameter control and internal quality of this boule were excellent.

Several other compositions were prepared for a second time because of the amount of material needed, size, or quality considerations. Among these runs was an additional trial on a 10% Tm, 0.5% Cr composition. The new run was NC-69 and gave an excellent boule using the 0.6mm/hr pull rate and 30 rpm. One further attempt was made with the 5% Eu composition. This run was NC-70 and it gave a good crystal with no banding along the length. These experiments completed the easiest of our growth runs and all of them were fairly straightforward. The average growth cycle for each boule was about 14-20 days depending on the respective pull rates.

3.3 Trials on U^{3+}:YAG

The incorporation of actinide elements into the garnet structure has not been considered seriously. In fact Geller[6] in his review up to 1967 does not even describe or mention the possibility of these particular elements. Yet there are many actinides which can form trivalent ions, are stable, and under many circumstances give a marked resemblance in their spectra to corresponding rare earths. Thus compare U^{3+} and Nd^{3+}, Pu^{3+} and Sm^{3+}, or Am^{3+} and Eu^{3+}. From a size viewpoint most of the trivalent actinides are no larger than La^{3+}. The latter cannot be completely substituted in garnets but certainly up to fraction as high as 0.4-0.5 formula unit. This fraction is well above the substitution levels generally employed in any laser crystal. Thus from a chemical and size viewpoint, it appeared that U^{3+} should fit within a garnet.

The type of dopant compound, growth atmosphere, and other conditions partially determine how and in what quantity U^{3+} may be incorporated in a crystal. Uranium forms several hydrated oxides of the type $UO_3 \cdot nH_2O$ and $UO_4 \cdot nH_2O$. In addition there are oxides of the UO, UO_2, U_2O_5, U_2O_7, U_3O_8, and UO_3 type. There are also various ordered phases of intermediate composition. For our first growth trials we chose to work with the UO_2 composition for a dopant, employ a reducing atmosphere of H_2, and assume a distribution coefficient similar to that for La (about 0.1).

The first run was NC-72 and a reasonably good crystal was grown at 0.6mm/hr and 30 rpm. The growth atmosphere consisted of a mixture of 97% N_2 and 3% H_2 by volume. In the past this gas mixture has led at times to a very brittle crystal. However no cracks or other difficulties occurred. The crystal (Figure 13) was nearly colorless since a dopant level of 0.05% U^{3+} was desired. After growth and cleaning of the crystal surface, a check was made under a UV lamp for fluorescence. No indication was given. The presence of radioactivity was determined by means of a scintillation counter but the response was almost indentical with the background. Thus we were not certain that U^{3+} was incorporated. A 2mm slice of crystal from the boule was polished on both ends and an absorption spectra was run in a spectrophotometer. Very little absorption above that of pure YAG was found so it was assumed that not much U^{3+} was in the crystal. A new charge was prepared for the crucible and another crystal was grown in a 0.1% H_2 atmosphere with similar growth conditions. This run was NC-73 and a very good crystal was obtained. A sample of the material was tested as before but no good evidence for the presence of U^{3+} in YAG was obtained. No further work was attempted on this system. It was thought that much higher levels of U should be used since the distribution coefficient was much lower than assumed. Obviously this problem was more involved than any other crystal growth run and not enough time could be spent on it under the

under the available funds. It does appear possible to incorporate small amounts of actinides in garnets but the chemistry is complex.

3.4 Growth of Nd:YAG

Under this program there were several requests for finished rods of Nd:YAG. Since these crystals were produced routinely at Airtron in production quantities no further description of growth is felt to be necessary. The complete account has been described in previous reports[7] on the growth and processing of high quality rods. The desired materials had several deviations from the ordinary. First the amount of Nd was adjusted. This ran from a low of 0.6 atom per cent to a high of 1.1 per cent for a grown boule. Finally the processing steps required different end face configurations in place of the normal flat-flat. Some of the intended experiments were to be carried out with the 1.30 μm laser line in place of the normal 1.06 μm. All of the above rods were delivered immediately with no problems.

3.5 Summary of Growth Runs

The main efforts of this program in crystal growth are outlined in Table I where the run numbers, crucible sizes, doping elements, doping concentrations, and boule sizes are given. Table II gives a summary of the delivered materials, run origin, the type or size of rod, coating requirement, and delivery number. Table III is an additional description of

Table I

Summary of Crystal Growth Runs

Run No.	Crucible Diam. (in.)	Doping Element	Doping (atom. %)	Crystal Results (diam. x length) Size (in)
NC-50	2.5	Er	100	Crucible leak
NC-51	2.5	Er	2	1 x 7
NC-52	2.5	Dy	2	No crystal
NC-53	2.5	Dy	2	No crystal
NC-54	2.5	Dy	2	1.2 x 4.3
NC-55-1	2.5	Ho	2	1.1 x 4.2
NC-55-2	2.5	Ho	2	1.0 x 3.9
NC-56	2.0	Er	100	0.9 x 3.8
NC-58	2.0	Ho	1	1.0 x 3.2
NC-61	2.5	Tm	5	0.7 x 1.1
NC-63	2.5	{Tm Cr}	10.0 0.5	0.8 x 4.2
NC-64	2.5	{Pr Cr}	0.5 1.0	0.7 x 2.8
NC-65	2.5	Yb	5	0.6 x 2.5
NC-66	2.5	Eu	3	0.8 x 4.0
NC-67	2.5	Eu	5	0.7 x 3.5
NC-68	2.5	Er	100	0.8 x 3.5
NC-69	2.5	{Tm Cr}	10 0.5	0.7 x 3.2
NC-70	2.5	Eu	5	0.6 x 3.3
NC-72	2.5	U	0.05	0.8 x 2.9
NC-73	2.5	U	0.05	0.7 x 2.2

Table II

Delivered Items of YAG

Growth Run	Composition	Items Delivered (mm)	Coated	Del. No.
NC-51	2% Er	Rod, 5 x 54	No	2
NC-51	2% Er	5 x 5 x 10	No	2
NC-54-1	2% Dy	Rod, 5 x 54	No	3
CZ-85	ZrO_2:Nd	Rod, 4 x 50	Yes	3
NC-55	2% Ho	Rod, 5 x 50	No	4
NC-54-1	2% Dy	5 x 5 x 10	No	4
4909	0% Nd	3 x 5 x 10	No	4
CZ-85	1% Nd	2 - 5	No	4
NC-61	5% Tm	5 x 5 x 10	No	6
NC-58	1% Ho	Rod, 5 x 54	No	7
NC-55	2% Ho	5 x 5 x 10	No	8
NC-65	5% Yb	Rod, 5 x 50	No	9
NC-56	100% Er	Rod, 3 x 30	No	10
NC-56	100% Er	Rod, 3 x 54	No	10

Table II (Continued)

Growth Run	Composition	Items Delivered (mm)	Coated	Del. No.
NC-66	Eu	Rod, 5 x 54	No	10
NC-72	0.05% U	Rod 5 x 54	No	10
NC-63	10% Tm 0.5% Cr	Rod 5 x 54	No	10
NC-63	10% Tm 0.5% Cr	Rod 5 x 54	Nc	10
NC-64	0.5% Pr 1.0% Cr	Rod 5 x 54	No	10
NC-58	1% Ho	5 x 54	No	10
NC-64	0.5% Pr 1.0% Cr	5 x 5 x 10	No	10
NC-67	5% Eu	5 x 5 x 10	No	10
NC-73	.05% U	5 x 5 x 10	No	10
NC-65	5% Yb	5 x 5 x 10	No	10
NC-51	2% Er	Rod, 2 x 21	Yes	11
NC-63	10% Tm 0.5% Cr	Rod, 2 x 21	Yes	11
NC-69	10% Tm 0.5% Cr	Rod, 2 x 21	Yes	11
NC-56	100% Er	Rod, 2 x 21	Yes	11
NC-68	100% Er	Rod, 2 x 21	Yes	11

Table III

Nd:YAG Rods Delivered

Rod No.	Size (in.)	% Nd	End Configuration	AR Coated	Del. No.
13221	0.25 x 2.50	0.6	Flat-flat	Yes	1
14271	0.25 x 3.00	1.1	Brewster	No	3
15396	0.25 x 3.00	1.1	12° off [111]	No	5
21008	0.25 x 3.00	1.1	Flat-flat	No	10
21009	0.25 x 3.00	1.1	Flat-flat	Yes	10
21010	0.38 x 3.00	1.1	Flat-flat	Yes	10

the Nd:YAG materials which were prepared and what rods were manufactured. Table IV lists the specifications of coatings that were suggested for certain rods to be tested at designated infrared wavelengths. These coatings were not developed or manufactured at Airtron but were subcontracted to the Optical Filter Corp. of Natick, Mass.

4.0 Conclusions

The development of satisfactory laser materials requires a cooperation of crystal growers and laser physicists. Unfortunately this arrangement is difficult to achieve within a single laboratory at the present time. With the specific design goals of laser materials which may operate in the 1-5 μm range, we have grown about twenty compositions of doped YAG crystals. These materials were fabricated into laser rods for pumping by flashlamps. Other configurations were developed and prepared for pumping by other lasers. All materials were delivered to the Naval Research Laboratory for examination and active testing under their internal physics programs. The delivered rods attained a quality equal to that of commercial samples. The boule which were grown gave no problems except for a proposed U^{3+}:YAG composition. Persistent efforts could solve the doping if necessary.

Table IV

Special Rod Coatings

Run No.	Composition	Coating Description
NC-51	2% Er	AR coated for 3.4μm
NC-51	2% Er	100% R at one end, 0.4% T at other end for 3.4μm; low R at 0.55, .86, 1.25, 1.70μm.
NC-63	10% Tm, 0.5% Cr	AR at 2.40 and 4.1μm
NC-63	10% Tm, 0.5 Cr	100% R at one end, 0.4% at other end for 2.4μm; low R from 2.4-4.4μm.
NC-56	100% Er	AR for 2.94μm
NC-56	100% Er	100% R for one end, 0.4% T at other end for 2.94μm

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